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UNITED STATES
DEPARTMENT OF THE INTERIOR
J. A. KRUG, SECRETARY

BUREAU OF MINES
R. R. SAYERS, DIRECTOR

INFORMATION CIRCULAR

REPORT ON THE INVESTIGATION BY FUELS AND LUBRICANTS TEAMS AT THE WINTERSHALL A. G., LÜTZKENDORF, NEAR MÜCHELN, GERMANY

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H. HOLLINGS

FOREWORD

In the early part of 1945 as the Allied armies advanced, German laboratories and plants became available for investigation. Since German development work had been hidden from the eyes of the world by the Reich's national policy of secrecy, and by 5½ years of war, it was obvious that much might be learned from such investigation. In particular, this was true in the synthetic-fuel industries deriving oil, gasoline, and a wide variety of chemicals from coal, for owing to the scarcity of domestic petroleum, Germany had been forced into intense development of this field in contrast to the limited amount of similar development by the United States and Great Britain. Before the fighting ended in Europe, the United States and Great Britain organized teams comprised of experts in all fields to investigate the research and industrial operations in Germany. The teams investigating coal, oil, gasification, and allied chemical fields included more than 30 American investigators and about an equivalent number of British investigators during 1945, and functioned with a reduced staff into 1946. German work inthe synthetic-liquid-fuels industries and allied fields was on such a large scale that even such extensive investigation could not possibly cover the subject in all detail. However, a great deal of information possible of direct application, and much information of a fundamental nature to help guide research work in this country for many years was uncovered. Studies of the German industry are continuing, and it is hoped that such gaps in the information as now exist will be filled in by future work.

The primary fields that were covered in the oil and synthetic-fuels investigation were concerned with petroleum refining and the gas-syntheses and coal-hydrogenation processes for producing oil from coal. The related fields of coal gasification, oxygen production, alcohol manufacture, lubricating oil production, and the production of waxes and edible fats, as well as a variety of other chemicals, were an inherent part of the investigation.

This report is one of a series which resulted from the investigations in Germany and other parts of Europe by the Technical Oil Mission, operating under the auspices of the Ministry of Fuel and Power for Great Britain and the Petroleum Administration for War and the Bureau of Mines for the United States. It is being published in accordance with the policy of the United States Government to make available to the interested public the results of the investigations of enemy research and industrial development.

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*Author of report.

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REPORT OF THE INVESTIGATIONS BY FUELS AND LUBRICANTS TEAMS AT THE WINTERSHALL A. G., LÜTZKENDORF, NEAR MÜCHELN, GERMANY

Reported by H. Hollings[1]

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[1] British Ministry of Fuel and Power.

INTRODUCTION

This report deals with the Schmalfeldt gasification plant for making synthesis gas, the Fischer-Tropsch plant, the hydrogenation plant and the catalyst factory, and includes a report on lubricating oil.

THE SCHMALFELDT GASIFICATION PLANT

The information was obtained on May 9 and 11, 1945, from Dr. Schneeberger (director), Dr. Schneider (director and power plant manager), Herr Dassau (manager of gas plant) and Herr Schultz (manager of Fischer-Tropsch plant). The plant was shut down as the result of very heavy air raid damage to other parts of the works.

The process consists of the gasification of brown coal in the entrained state. The designer, Dr. H. Schmalfeldt, lived at Lutzkendorf from the start-up in 1938 until 1940, and is now believed to be living in the Kassel area. Before this plant was built there had been an experimental unit at Rühlen, but so far as is known, there is no other large-scale installation.

The principles of the process are as follows. Brown coal was obtained from an open mine in the neighbourhood. Typical analyses were:-

As Received		Dry Basis	
H_2O	50-54%	C	60%
Ash	5-6%	H	4%
Tar	4-5%	O	18-20%
Total S	2-2.5%	S	3%
Volatile S	1-1.5%	N	1%
Calorific Value	2300-2400 k.cals per kg.	Ash	12%

The tar content was rather low for brown coal, and so the coal was not well suited to the normal treatment (carbonization to produce tar for hydrogenation, with use of coke in Winkler generators and in boilers). The raw coal was elevated and crushed in hammer mills to below 20 mm., dropping into a raw coal bunker, from whence it was fed by Redler screw conveyors through star feeders into the side of the bottom of the gas drier. The contents of the bunker were kept under nitrogen pressure. Recycled synthesis gas and steam at a temperature of 1,000° C. flowed upwards in the gas drier (see Fig. 2), and the sudden heating of the raw brown coal caused it to dry and decrepitate, the particles becoming entrained. The gas leaving the drier was passed through a classifier where large lumps of coal were removed, to be crushed and returned to the drier. The gas next passed to a large cyclone separator, and was then divided into two streams, one of which constituted the synthesis gas make while the other was recycled. On each stream there was a small cyclone followed by a washer. The dry coal dust separated in the cyclones was used in the gasifiers (gas generators) and in the producers. The recycled synthesis gas was passed to a hot generator where it was heated to 1,300°C., and then passed through two gasifiers or generators in series, to complete the cycle by entering the drier. Dry coal dust was injected into the top of the first generator and was gasified.

The heat of reaction was originally supplied by burning producer gas in one of two regenerators, used alternately, the heat being stored in chequer brick until given up to a mixture of recycled synthesis gas and steam. The gasification plant was intended to supply synthesis gas for the Fischer-Tropsch plant (designed to produce 75,000 tonnes of crude oil per annum), but later a hydrogenation plant was added (designed to produce 50,000 tonnes per annum) and the gas requirement was increased. In order to make more gas, additional heat had been introduced by adding oxygen to the recycled gas. The original designed output per gasifier unit (considering the two generators in series as one gasifier unit) was 20,000 cubic meters per hour (measured at 0°C. and 760 mm.), but in fact the output obtainable without the use of oxygen was only 15,000 cu.m. per hour. When using oxygen, the maximum output was 30,000 cu.m. per hour, although more usually 20-25,000 cu.m. per hour were made.

The producer gas was made by gasification of entrained dry coal dust, but there was no recycling of gas or use of oxygen. Air, steam and dry coal dust were fed into a tower, and the sensible heat in the exit gases was abstracted by waste heat boilers. The maximum output of a producer was 30-35,000 cu.m. per hour.

The plant consisted of four gasifier units each, as shown in Figure 2, and five producers, and usually one of each was out of action for cleaning. The plant was very spacious considering its capacity, although it was made up of relatively simple pieces of equipment. The four gasifier units (2 generators, 2 regenerators, drier, with stocks and washers) occupied an area of about 100 m. x 30 m., the vessels being 20-24 m. high. The gas boasting house was outside this area, while the producers occupied a separate site. Figures 1A and 1B show a rough layout and arrangement of a synthesis gas unit, while Figure 2 is a diagrammatic representation of the flow. Figure 3 is a photograph of one end of the synthesis gas plant. Figure 5 is a rough layout of a producer gas unit, and Figure 6 is a flowsheet for producer gas. The individual items of plant will now be considered in turn.

The gas drier was a vertical brick-lined chimney, 1.2 m. internal diameter and 22 m. total height. The crushed raw brown coal dropped into the drier without any conveying gas, at a point 15 m. below the top of the drier. Two inlets were available, one working and one spare; each was steam-heated to prevent sticking of the coal, but was brick-lined near the drier itself. The feed pipe was at an angle of about 30° to the vertical. No special mixing of coal and gas was attempted; the high turbulence of the gas, which had just passed through a bend of about 2.5 m. radius of curvature, together with the explosion of each particle, was sufficient to give good mixing. The final temperature at the top of the drier was 200-300°C. At full output, the dry gas rate entering the drier is calculated to be about 90,000 cu.m. per hour, so that taking into account the steam present, calculated as 91,500 cu.m. per hour, the average gas velocity in the drier

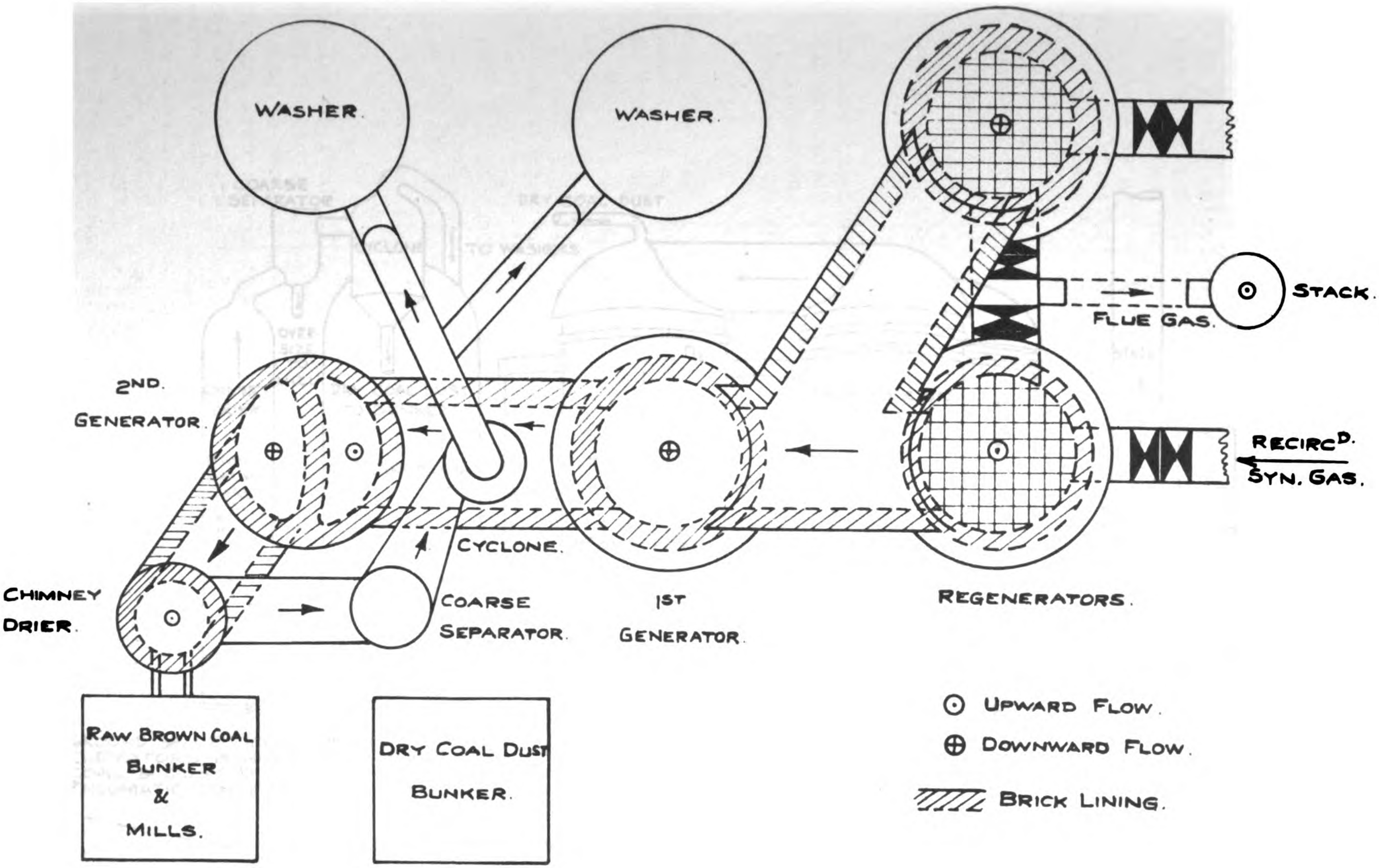

FIG. 1A. PLAN VIEW & ROUGH LAYOUT OF SYNTHESIS GAS UNIT.

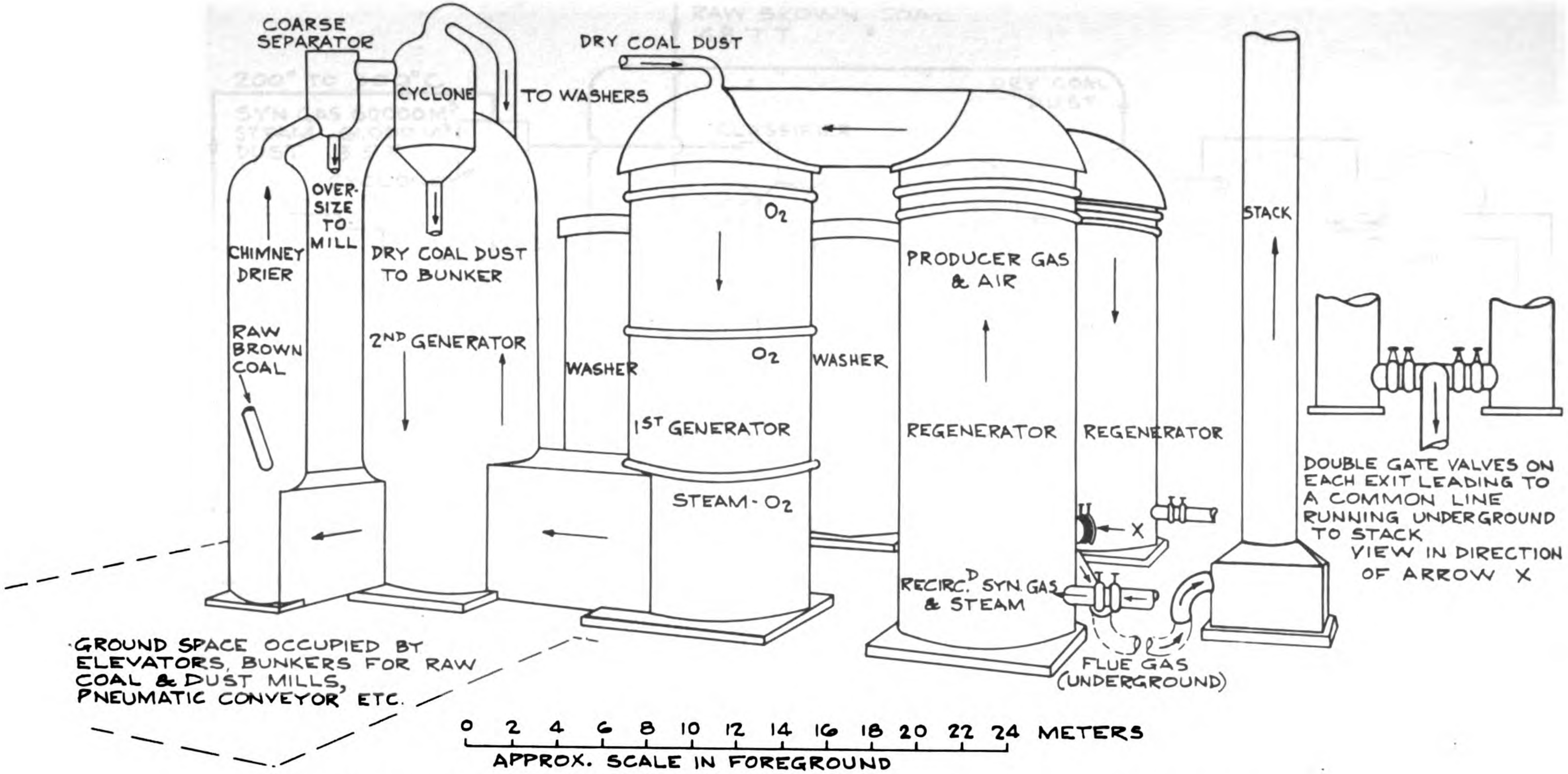
COARSE SEPARATOR
CYCLONE
TO WASHERS
DRY COAL DUST
OVER-SIZE TO MILL
CHIMNEY DRIER
DRY COAL DUST TO BUNKER
RAW BROWN COAL
2ND GENERATOR
WASHER
O_2
O_2
1ST GENERATOR
STEAM - O_2
WASHER
PRODUCER GAS & AIR
REGENERATOR
REGENERATOR
STACK
X
RECIRC.D SYN. GAS & STEAM
FLUE GAS (UNDERGROUND)
DOUBLE GATE VALVES ON EACH EXIT LEADING TO A COMMON LINE RUNNING UNDERGROUND TO STACK
VIEW IN DIRECTION OF ARROW X
GROUND SPACE OCCUPIED BY ELEVATORS, BUNKERS FOR RAW COAL & DUST MILLS, PNEUMATIC CONVEYOR ETC.
0 2 4 6 8 10 12 14 16 18 20 22 24 METERS
APPROX. SCALE IN FOREGROUND

FIG. 1B.- SYNTHESIS GAS UNIT (SIMPLIFIED BUT ROUGHLY TO SIZE-BUNKER BUILDING SHOWN REMOVED).

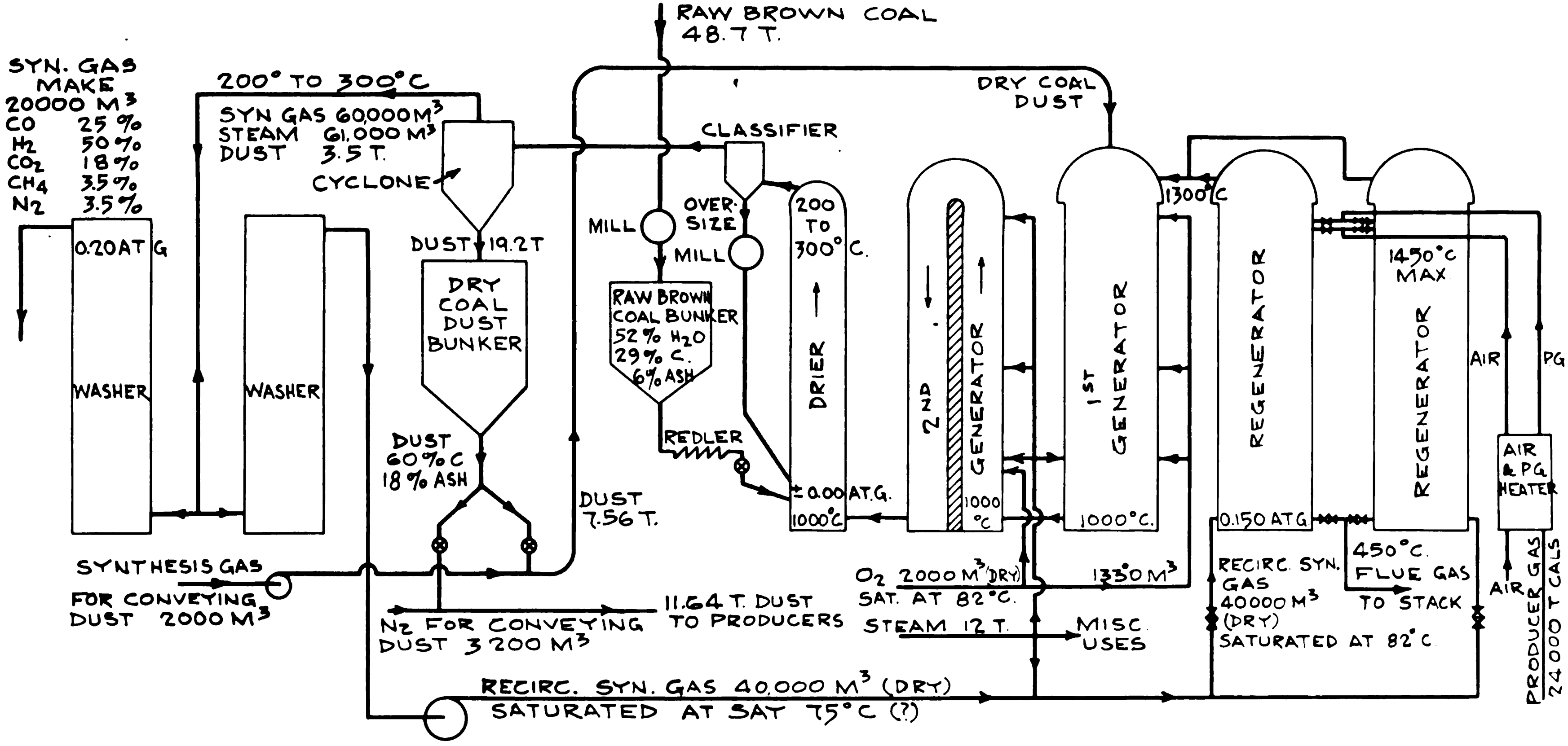

FIGURE 2.- FLOWSHEET FOR SYNTHESIS GAS UNIT MAKING 20,000 M^3/HR. - MAX. OUTPUT 30,000 M^3/HR.

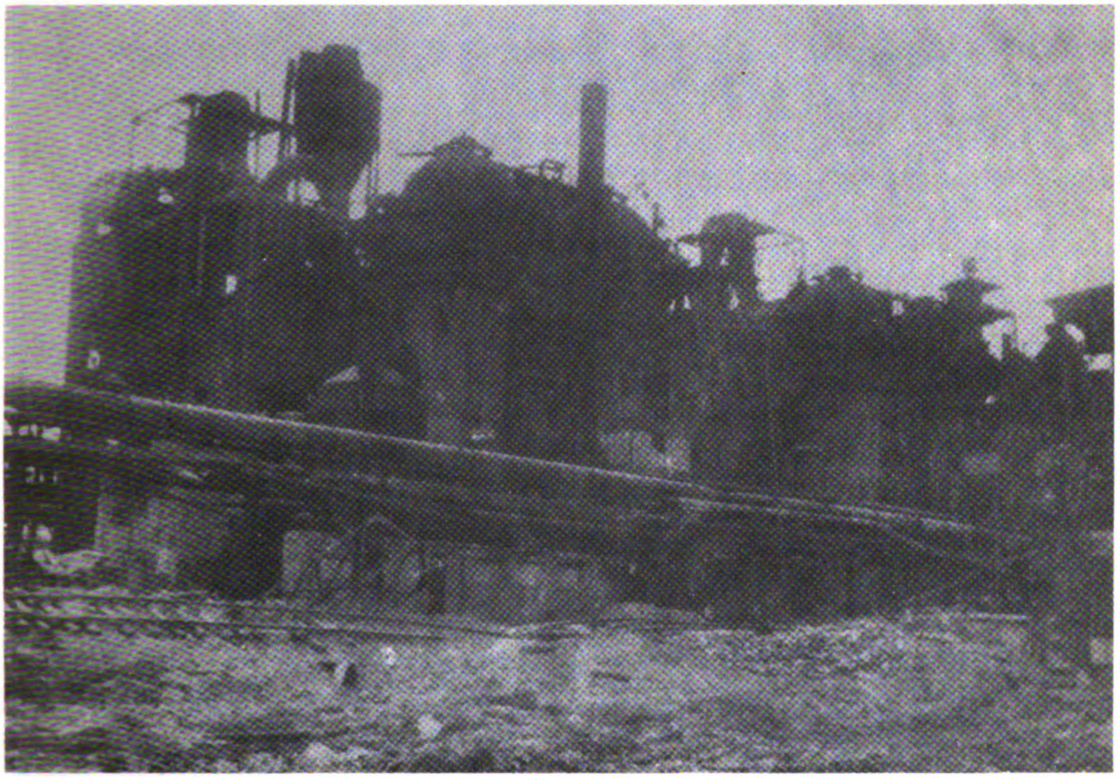

Figure 3. - One end of the synthesis gas plant.

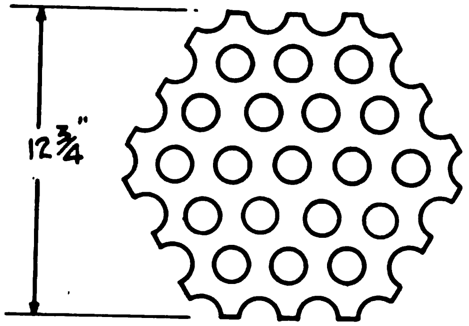

FIGURE 4A.- PLAN OF DIDIER BRICK FOR REGENERATORS.

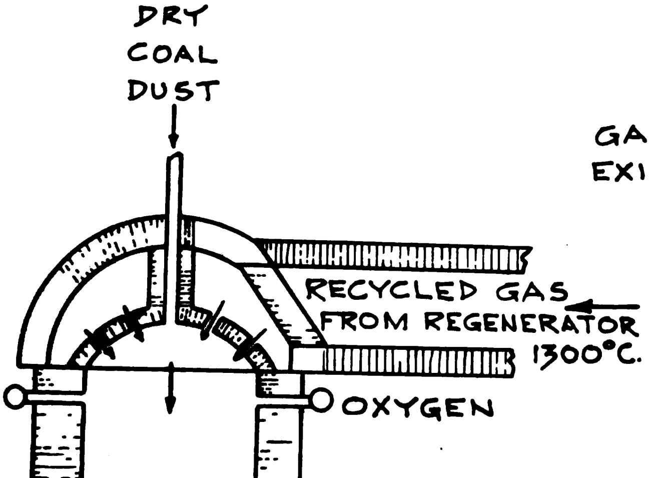

FIG. 4B.- TOP OF FIRST GENERATON (SECTION)

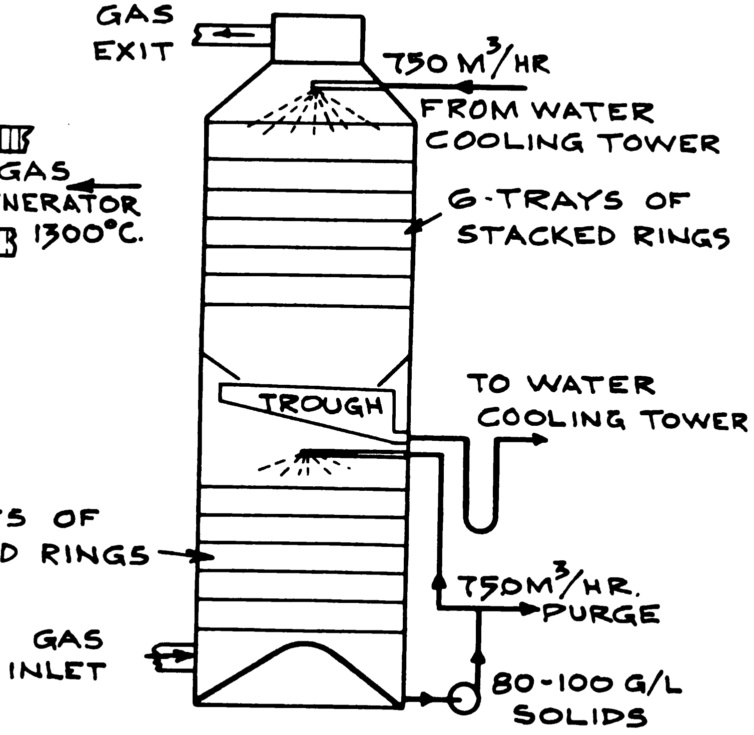

FIG. 4C.- SECTION OF WASHER.

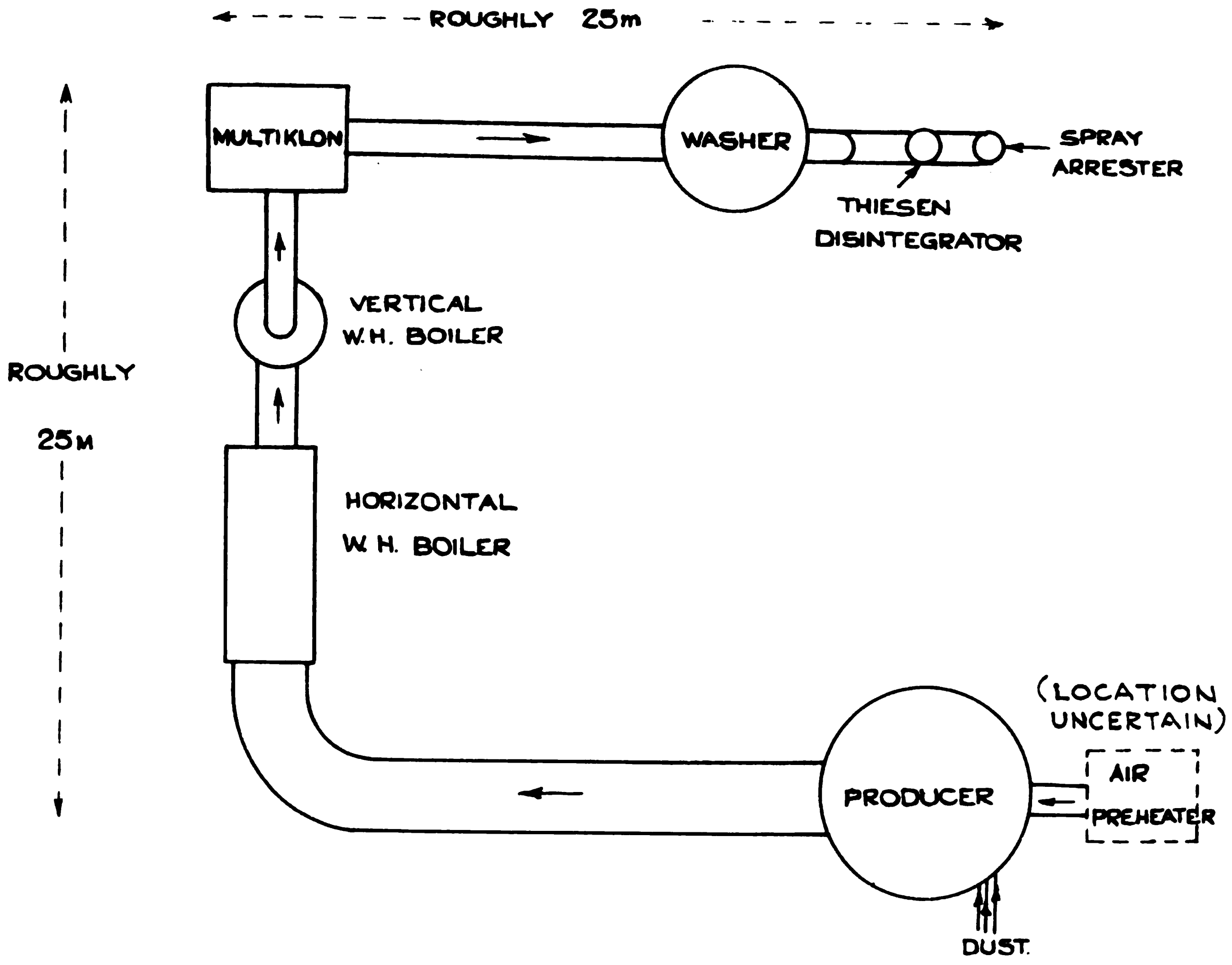

FIG 5 ROUGH LAYOUT OF RRODUCER GAS UNIT

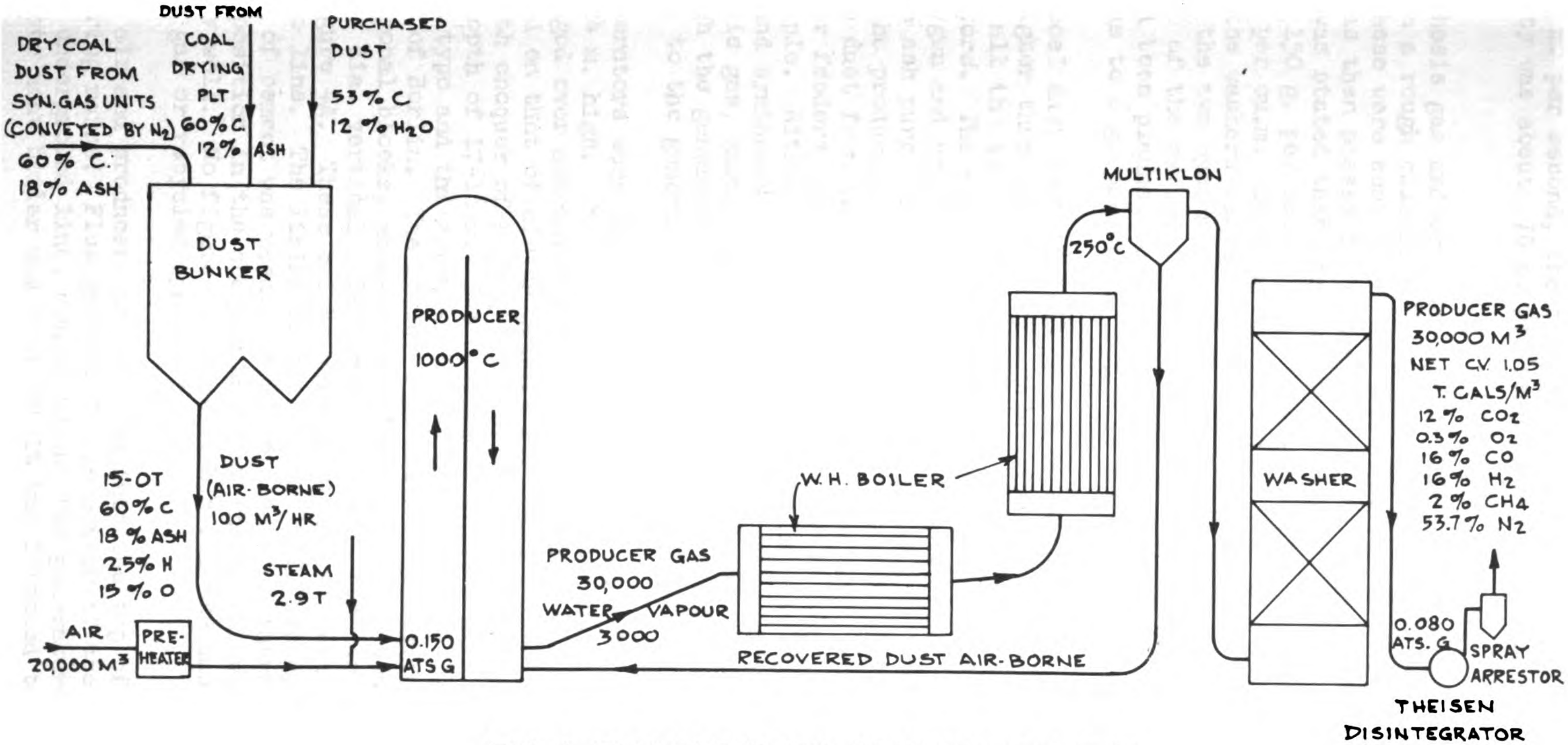

FIG. 6. FLOWSHEET FOR PRODUCER GAS UNIT

Recycled synthesis gas, saturated with water vapour at 82°C., entered the bottom of the regenerator and left it through the cupola at the top at 1,300°C. on its way to the first generator. The maximum brickwork temperature was 1,450°C. at the top; the average exit flue gas temperature was 450°C. Double isolation valves were used on each flue gas and recycled gas line, with the portion between the valves automatically vented to atmosphere when the valves were shut. The isolation valves on the air and producer gas were not seen, but may also have been double. Details were not obtained of the method of isolating the top of a regenerator while heating it on alternate cycles; it is possible that no valves were used, reliance being placed on pressure control to prevent more than a slight flow of synthesis gas into the regenerators. At first there had been trouble with erosion at the top of the cupola, but this had been cured by constructional changes aimed at making the linear velocities of the gas in the cupola and off-take pipe more nearly equal.

The first generator was a brick-lined vessel, 5.5 m. int. dia. by 24 m. high. The special design of the cupola, with its false roof, is shown in Figure 4B. Dry coal dust, conveyed by synthesis gas at 2-1/2 atm. pressure, was fed down through a passage in the center of the cupola. The hot recycled gas and steam mixture from the regenerator was fed through ports in the false roof. Oxygen, saturated at 82°C., was introduced from ring mains through ports near the top and middle of the generator; near the bottom steam as well as oxygen was admitted to avoid slagging. The temperature fell from 1,300°C. at the top to 1,000°C. at the bottom.

The oxygen was supplied by three Linde-Fränkl units, two working and one spare, each producing 4,000 cu.m/hr.

The second generator was a brick-lined vessel, 5.5 m. int. dia. by 24 m. high. On three units it was divided internally by a vertical wall, but on the fourth (and latest) unit it had no such division wall. The division wall was shaped as shown in Figure 1A, the two portions having approximately the same area of cross section. In the unit with no division wall the gas was brought down to the bottom of the drier by an external pipe. One third of the total oxygen used was added near the bottom inlet of the generator, while steam was added as required at various points, the aim being to maintain the temperature at 1,000°C. It was stated that the division wall was not necessary; the unit without it worked just as well. It was further stated that the second generator was not needed at all except for high outputs, such as were obtained by the use of oxygen.

From the data given, it is calculated that the gasification period (time of contact of coal in generators) was 4.5 seconds at 30,000 cu.m. per hour, and 6.0 seconds at 20,000 cu.m. per hour.

Each unit had two washers, one for recycled gas (50-60,000 cu.m. per hour) and one for synthesis gas (25,000 cu.m. per hour). The washers were the same size and were fed with water at the same rate, despite the differences in gas load. A typical arrangement is shown in Figure 4c. Each washer was about 6 m. diam. by 22 m. total height. The washer was divided into two sections with separate water circulation systems, each section being packed with 6 trays of stacked 80 mm. spiral Raschig rings. About 750 cu.m. of water per hour were circulated through the bottom section to remove the bulk of the dust. A purge was taken from this system to keep the concentration of solids at 80-100 g. per liter. 750 cu.m. of water per hour were circulated through the top section and through a water cooling tower. The high water rates were necessary to prevent choking the packing with dust; but at each six monthly shut-down the rings were removed and washed, the work on 8 washers for the generator plant and 5 washers for the producers providing continuous employment for 14 day men.

The gas pressure at the bottom of the regenerator was 0.150 ats. gauge. Since the pressure at the point of entry of coal into the drier had to be kept very close to atmospheric, to prevent gas leaking back up the coal feed pipe, the drier and washer had to be run at a pressure below atmospheric. The pressure at the top of the washers was - 0.020 ats. gauge.

There were 5 producer units, each making at most 30,000 to 35,000 cu. m. of producer gas per hour, although normally making less. Each unit consisted of the producer followed by waste heat boilers, Multiklons, washtower and Theisen disintegrator. The producer was a brick-lined tower, 5 m. int. diam. by 24 m. high, with an internal division wall, very similar to the second generator. Dry coal dust was blown into the bottom of the tower with steam and air, the mixture passing up one side and down the other. The maximum temperature reached was 1,000°C. The gasification time was about 11 seconds when making 30,000 cu.m. per hour. The dry coal dust (18% ash, 60% C) was conveyed from the generator units to a bunker by means of nitrogen, but it was conveyed from the bunker to the producers by means of air. About 15 tonnes per hour were conveyed by 1,000 cu.m. of air per hour, through three pipe-lines each of 125 mm. int. diam. Assuming atmospheric pressure, this corresponds to a velocity of 7.5 m. per second and a dust content of 15,000 g. per cu.m. It was also stated that the dry coal dust fed to the producers amounted to 0.5 tonnes per 1,000 cu.m. of producer gas. Gases from the producers passed through a horizontal and a vertical waste heat boiler in series, which reduced the temperature to 250°C. Erosion at the inlet of the first boiler limited the running time of a unit to six months before overhaul. Multiklons followed the boilers, and the dust recovered was blown back by compressed air into the bottom of the producer.

The wash tower was 5-5.5 m. int. diam. by 22 m. high, filled with ordinary 2" Raschig rings. In design it was very similar to the washers on the synthesis gas, but the water rate was only 500 cu.m. per hour. It was said to be too small and to pass too much dust. A Theisen disintegrator removed most of the remaining dust, and after passing through a spray arrestor, the gas was pumped into the factory fuel gas system. The amount of gas used for heating a single gasification unit was 24,000 cu.m. per hour, consisting of 3,000-4,000 cu.m. of rest gas from the Fischer-Tropsch plant (2,400 Kcals/cu.m.) and 21,000-20,000 cu.m. of producer gas (1,075 Kcals/cu.m.)

The pressure was 0.150 ats. gauge at the bottom of the producer and 0.080 ats. gauge before the Theisen disintegrator.

Three of the boilers installed in the power plant could use only dry coal, and a plant fired by coal and producer gas was installed to provide this dry coal. This plant was also used to a certain extent to supply dry coal to the producers, when for any reason the driers of the synthesis gas units were unable to provide enough coal. A small quantity of dried brown coal dust, containing 12% H_2O and 53% C, was also purchased and used partly on the boilers and partly on the producers.

The content of dust in the synthesis gas was stated to be reduced to 30-40 mg. per cu.m. by the water washer, and to 23-25 mg. per cu.m. by the Theisen disintegrator. There was a further water wash before the synthesis gas passed to an Alkazid plant for removal of most of the hydrogen sulfide. There was stated to be no difficulty due to dust in the Alkazid plant.

Ash and slag gradually accumulated in the first generator, and when oxygen was used, the generator had to be cleaned out every six months, whereas if no oxygen was used, the plant could be run for 18 months. During these shut-downs, other maintenance work was carried out. As a rule, a shut-down lasted 42 days - 14 days to cool down, 14 days to carry out repairs, and 14 days to heat up. The ash and slag mixture was white and very hard, and had to be chiselled out.

The composition of the synthesis gas was stated to be:-

	Without Oxygen	With Oxygen
CO %	28	25
H_2	56	50
CO_2	10	18
CH_4	3	3.5
N_2	3	3.5

Thus no conversion was required before synthesis. The gas composition could be adjusted by varying the conditions of gasification and the amounts of steam and oxygen.

The composition of the producer gas was as follows:-

CO	16 %
H_2	16
CH_4	2
CO_2	12
O_2	0.3
N_2	53.7

The plant records for 1943 gave the following figures:-

Synthesis gas made	330,774,000 cu.m.
Producer gas made	522,633,000 cu.m.
Raw coal direct to gas production	308,593 tonnes
Raw coal to coal drying plant and thence to producers	119,058 tonnes
Purchased coal dust to producers	7,076 tonnes
Synthesis gas composition	$CO + H_2$ 74.7% H_2/CO 1.98

The results for April 1944 were given as:-

Synthesis gas		45,900,000 cu.m.
Producer gas made		47,950,000 cu.m.
Raw coal direct to gas production		111,823 tonnes
Raw coal to coal drying plant and thence to producers		1,012 tonnes
Purchase coal dust to producers		2,454 tonnes
Synthesis gas composition	$CO + H_2$ 75.2% H_2/CO	2.0
Oxygen used		4,388,600 cu.m.
Nitrogen used for conveying dust		7,193,600 cu.m.
Power for synthesis gas production		1,152,000 kwh.
Power for producer gas production		1,564,000 kwh.
Steam to synthesis gas plant excluding producers		27,375 tonnes
Steam to producers		4,622 tonnes

The labour requirements of the plant were as follows:-

Process:	Synthesis gas	180 men
	Producer gas	80
	Coal transport and preparation	70
		330
Maintenance:	Fitters and labourers	80
	Bricklayers	10
	Electricians	5
	Instruments	5
		100

GAS PURIFICATION

The synthesis gas contains 17-30 grams of hydrogen sulphide per cu.m., and the bulk of this was removed in an Alkazid plant, the hydrogen sulphide being converted to sulphur of 99.6-99.9% purity in a Claus kiln. The gas also contained 6.8 g. of benzole per cu.m. and was next washed with oil. The remaining hydrogen sulphide was removed in iron oxide boxes in which Lux was used.

The gas then contained 40-100 g. of organic sulphur per 100 cu.m., of which as much as 20% was in the form of thiophen as compared with 10% in coke oven gas. Gum-forming substances were also present. The high content of thiophen and gum-forming substances was ascribed to the characteristic feature of the Schmalfeldt process, where drying and gasification were carried out in the same apparatus. It was considered that the high content of thiophen and gum formers would not be found if the drying were carried out first, for example in a Büttner drier. Furthermore, it might be expected that these substances would be destroyed in passing through the regenerator at 1,300°C., but the system was such that one-third of the gaseous products from the drier was drawn off in the make and did not pass through the regenerators or generators. Similarly one-third of the products from the generators did not pass through the regenerators.

The gas was passed through 70% luxmasse with 30% soda at 160-280°C. for removal of organic sulphur compounds. The gas so purified gave considerable difficulty in the Fischer-Tropsch process, which used a cobalt-kieselguhr catalyst. The gum formers and possibly a little dust still present interfered with the activity of the catalyst, but the main difficulty was thought to be poisoning of the catalyst by sulphur. There was no difficulty in using the gas for hydrogenation, where sulphur is not a poison. In an attempt to increase the efficiency of purification, an active carbon plant had been installed before the hot luxmasse, in the belief that it would remove the gum formers which (apart from their action on the Fischer catalyst) were thought to reduce the activity of the hot luxmasse as a sulphur purification material. The installation of the active carbon plant reduced the organic sulphur content of the gas leaving the hot luxmasse from 1.5-2.5 g. per 100 cu.m. to 0.3 g. per 100 cu.m., which was a satisfactory figure. The plant was run with this improvement for only two months before bombing stopped work, but during this period, the results were promising. It was stated that it had been intended as a further improvement to use a special sulphur removal type of active carbon instead of the benzole recovery type.

FISCHER-TROPSCH PLANT

The following information was obtained mainly from Herr Schültz on 11 May 1945.

There were 2 catalyst chamber buildings with 144 chambers altogether. These were generally worked in two stages, but sometimes in three stages. Conversion in the first stage was about 50%. The division between the two stages was variable and dependent upon gas purity. During the period when the gas was bad, the first stage was largely the means for purifying the gas and was operated with a throughput of 1,500 m^3 per chamber per hour instead of the normal 800-900 m^3 per hour.

The most recent yields after difficulties with the gas had been overcome were 115-118 gm. per Nm^3 Idealgas. In 1940 and 1941, a yield of 130 gm. per Nm^3 was attained by changing the catalyst every 1-1/2-2 months. The catalyst chambers were started up with 5-1/2 atm. on the steam side and ultimately went up to 15 atm. (200°C.). The catalyst was extracted with solvent in situ every four weeks. It was only hydrogenated in special cases. The catalyst chambers were heated with steam at 19-20 atm. for starting.

For the last 1-1/2 years they had used catalyst obtained from the adjacent catalyst factory, which was owned 50% by Ruhrchemie and 50% by Wintershall. The catalyst was obtained from the catalyst factory in the unreduced stage and was reduced with hydrogen from the hydrogenation plant. Reduction was carried out at 400°C. The hydrogen was dried by ammonia cooling and silica gel to less than 0.1 gm. per m^3 moisture. The hydrogen used contained 94% H_2, the remainder being N_2 and CH_4. The circulating hydrogen contained about 1/2% CO_2 but no CO.

The production of the plant was at the rate of about 30,000 tons per year without Gasol, during the short period when good gas was available before the bombing. They were in the process of increasing production when the bombing took place.

The plant included a Carl still cracking plant which had never been worked owing to a change in the policy of dealing with the products.

CATALYST FACTORY

The following information was obtained mainly from Dr. Joswig on 11 May 1945.

The particular feature of this plant was that they claimed a very high recovery of cobalt in the catalyst regeneration. The departures from the normal process which led to this result were due to the restrictions on emitting effluent from the factory, which necessitated re-using water and evaporating the final effluent.

The spent catalyst, from which the wax had been extracted, was ground with wash water from the following stage. This wash water contained about 5 gm. per liter of Co. It was then extracted with boiling nitric acid (50%) in vessels of about 30 m^3 capacity. It was then filtered on a filter press and washed. The first wash water was mixed with the filtrate. The second wash water was used for grinding the incoming spent catalyst, as mentioned above. The third wash water was used for the first wash of the next batch. It was claimed that the loss of Co in the filter pressing operation was less than 0.1%. Joswig claimed that they had achieved an overall loss in the factory of only 0.3% compared with 2% at Ruhland and 4-5% at Holten.

The Co solution was then treated with soda to precipitate Fe, Al and Th. The sludge obtained by filtration (Thoriumschlamm) was sent to Ruhland. Sodium fluoride was then added to remove calcium. To ensure complete removal of calcium, an excess of sodium fluoride was added which precipitated part of the magnesium. The liquor was then filtered using kieselguhr as a filter aid. The filter cake was washed twice, the first washings being added to the filtrate and the second washings being used for the first wash of the next batch. The Co concentration was finally 40-45 gm. per liter. This was sometimes strengthened with the fresh Co added as make-up. When fresh Co was used, it was dissolved in nitric acid and treated with a small amount of soda and then filtered before mixing with the recovered Co solution.

The Mg and Th were then added to solution. After precipitation, kieselguhr was added. The filtrate from the precipitated catalyst contained about 30 gm. per liter of sodium nitrate. This figure was achieved by reducing wash waters. The filtrate was then treated with soda and settled to recover any Co which had passed through the filters. After settling, the solution was evaporated in a triple effect evaporator to recover sodium nitrate and produce distilled water for the catalyst manufacture. In the second evaporation stage, some caustic soda was added to ensure complete precipitation of any residual Co which was filtered out before the third stage. Joswig stated that an appreciable amount of Co may remain in solution as bicarbonate.

The catalyst sludge from the presses was mixed with water and with the dust screened out from the dried catalyst. The mixture was then filtered on a rotary vacuum filter. The filter cake was put through an extruder with 6 mm. holes and then passed to a Büttner turbo-drier. The dried catalyst was screened to give a product of 1-3 mm. The dust was mixed with the new precipitate, as mentioned above. The over-size was returned to the screens and gradually broken down to the required size.

The composition of the catalyst was given as:-

100	parts	Co
10	"	MgO
5	"	ThO_2

The catalyst was made up to give 800-900 kg. Co per chamber, using an appropriate amount of kieselguhr depending on its density. From 1.7 to 2.3 tons of kieselguhr were used per ton of Co.

The capacity of the plant was rated at 100 catalyst chamber fillings per month, but the production had been only 60 owing to limited demand. The plant started up in September 1941 but only came into operation in April 1942. Lützkendorf made catalyst for the adjoining F.-T. plant and also for the one at Odertal (Deschowitz).

The reason for producing unreduced catalyst at Lützkendorf and reducing it at the F.-T. plants was not very clear. The division of responsibility between the two stages seems undesirable. One reason that was given for adopting this procedure was that the F.-T. plants had pure hydrogen available as they require it for the hydrogenation in the catalyst chambers. It is possible that freshly reduced catalyst may be better than reduced catalyst which has spent some time in storage or in transit. It may be conjectured that the procedure was at least partly dictated by military considerations. If it were desired to maintain a substantial storage of manufactured catalyst against possible destruction of the catalyst manufacture, storage of the unreduced catalyst would be a much simpler problem than storage in substantial quantity of the reduced catalyst.

HYDROGENATION PLANT

The following staff were seen on 11 May 1945 and 17 May 1945 in connection with the hydrogenation section:-

Dr. Schnesberger	Managing Director.
Dr. Wielland,	Hydrogenation Manager.

The following information was obtained from interrogation and also from the notes of members of the C.A.F. team.

GENERAL.

The hydrogenation plant was built to hydrogenate the residues from the lubricating oil plant in conjunction with Bituminous Coal Tar. The planned output was 50,000 Tnes/year, but the best achieved was 42,000 Tnes/year in 1944.

The plant consisted of one liquid phase stall and one vapour phase stall, both of which operated at 700 ats. The catalyst in the vapour phase stall was 6434.

The Bituminous Coal Tar came from Mahrisch Ostrau and was distilled with the product from the liquid phase stall to cut into two fractions, the cutting point being 350° C. (330° C. ?). The lighter fraction was passed over the vapour phase catalyst under straight through conditions when making petrol and diesel oil and under recycle conditions when making petrol only.

HYDROGEN SUPPLY.

The hydrogen from the hydrogenation plant came from the Wintershall generators. The raw water-gas was passed through an 8 ats CO conversion plant and the Co_2 washed out at the same pressure. The main compressors therefore had a suction pressure of 8 ats and delivered to 700 ats direct. The CO removal plant worked at 200 ats. after the third stage of the compressors. Another stage sufficed to raise the pressure to 700 ats.

The gas fed to the hydrogenation plant had a hydrogen purity of only 91-92%, which is very poor by normal standards. They had only achieved this purity by the installation of the CO conversion plant, before which they had worked with a make-up gas of about 86% purity.

For 1943 over a period of ten months, they had records of the following costs:-

Gas production	681,319	RM including 155,000 Rm for labour.
Gas boosting	24,306	RM.
H_2O removal	61,252	"
-- ---	6,023	"
Oil Scrubbing	26,143	"
Org. Sulph. Rem.	88,946	"
1/5 of Gen. Costs	19,000	"
	906,989	RM which amounts to 2.70 pf/m^3 Raw Water gas.

This is the price of raw water gas as supplied both to the Hydrogenation and Fischer-Tropsch plants. For the hydrogenation plant, the gas was further purified and the cost build-up for this section for the month of October 1943 is:-

Raw Water-Gas at 2.70 pf/m^3	222,220 RM
CO conversion and CO_2 rem.	75,480 "
Compression	50,000 "
CO removal	17,536 "
1/5th Gen. Costs	21,338 "
	386,582 RM which,

for 5,295,700 m^3 of 92% hydrogen, is 7,30 of pf/m^3.

CIRCULATION AND INJECTION PLANTS.

There were four circulators with an output of 70,000 m^3/hr. and one of 100,000 m^3/hr., all of them being electric driven.

They had two hot recycle pumps for the liquid phase stall of 10 m^3/hr. capacity, with four injectors having the same capacity for the injection of feed to the stalls. They also had four catalyst injection pumps which were steam driven and had a capacity of 0.25 m^3/hr.

LIQUID PHASE STALL.

This stall was originally built with two interchangers, two convertors, a hot catchpot, a gas-fired preheater, a cooler and a cold catchpot, with arrangements for the recycle of hot oil from the base of the hot catchpot to the inlet preheater.

There was also an emergency blow-down tank for the burden of the stall in case of emergency. Owing to poor make-up gas quality, bad operation by inexperienced staff and change of composition of the feed to the liquid phase stall from that from which the plant was designed, they had not achieved the rate and feed of reaction they had expected. They were therefore short of heating capacity in the preheater and could not maintain the temperatures in the convertors as easily as they would have liked. To overcome these running difficulties, they had installed a third interchanger, enlarged the fan capacity on the preheater, and had even applied a 1" thickness of lagging to the outside of the convertors in order to increase the heat input to the inlet reactants and to reduce the heat losses.

The feed to the stall was 8-12 m^3/hr. of oil with 100-150 liters/hour of catalyst paste made up as a paste of 40% solids from I.G. catalyst 10927 and vacuum residue from the Natural Oil section of the works. The running temperature was 470° C. and the reaction volume 10 m^3 (from dimensions) or 16 m^3 (interrogation) - more probably the former. The hot recycle rate was 6-10 m^3/hour. The catalyst was adjusted so that the H.O.L.D. from the hot catchpot was a clear colour and not darkened by the presence of Asphalts. The let-down from the hot catchpot in order to purge the solids entering with the catalyst paste was about twice the paste injection. The purge contained about 1.5-1.8% hard asphalts.

The H.O.L.D. was fugalled to give a residue which they put to the tip and a relatively clear oil of 0.2% solids which they sent to the liquid phase still. There was no gas washing plant to remove the gases made during the hydrogenation.

In the product from the cold catchpot they got 50% heavy oil which was recycled back to the stall. The product was distilled with the imported tars and the heavy oil portion fed to the liquid phase stall.

VAPOUR PHASE STALL.

The stall consisted of 3 convertors with 2 interchangers and a gas-fired preheater, a cooler and a cold catchpot.

The catalyst was the I.G. 6434 and the reaction volume would be about 13 m . When running on straight through operation they made a cold catchpot product containing 40% petrol and the remainder diesel oil. The petrol had a 74 O.N. with an FBP of 180° C. and the diesel oil a Cetane No. of 40-45 with a setting point of -40° C.

The gases made in the hydrogenation section were not treated to remove the liquifiable gases and the lean gases were sent to the fuel gas system and the rich gas to the Wintershall generators.

Overall hydrogen requirements are estimated at about 1,300 m^3/Tns of feed when making petrol and diesel oil.

HYDROGENATION RUNNING COSTS 1943.

Liquid Phase Injection & Circulation	3.85	RM/Tne of feed.
Vapour " " "	10.84	" " .
Liquid Phase Stall	17.17	" of Cold Catchpot Product.
Vapour Phase Stall	7.87	RM/Tne of Cold Catchpot Product
Total Distillation	45.96	RM/Tne feed.

In 10 months of 1943 they made 29,523 tonnes of Vapour Phase Cold Catchpot product at a cost of 7,448,456 RM., i.e. 255 RM/Tne. This is the running cost only and does not include any amortization or interest, etc.

LABOUR REQUIREMENTS.

Average figures for the early part of 1944 show the following:

Gas Production	500
Fischer Synthesis	245
Hydrogenation	280
O_2 Steam Elec.	50
Nat. Oil Section	200
Final Refining	110
Power Plant	270
Workshops	275
Construction etc.	200
Watchmen etc.	580

In addition to these, they had other workers whose exact location is not available from the field notes.

The total strength was made up of about 4,000 workers and 380 staff, but of the 4,000, about 1,000 were engaged on alteration and extension work, and not on production.

CAPITAL COSTS.

The split of capital costs were given as:-

Gas Plant -	22,610,614
Fischer Plant -	18,182,250
Hydrogenation -	18,606,851
Lube Oil -	9,488,452
Final Dist. -	6,092,751
Auxiliary -	5,033,343
Railways -	2,955,458
Water Supply -	7,000,994
Power Plant -	18,297,489
General -	9,138,176
	117,405,389

The engineer estimated that for every tonne of steel in the finished plant, they had had to spend about 1,660 RM.

LUBRICATING OIL

Specifications on gasolines and diesel oils manufactured at Lützkendorf.

The personnel interviewed at the plant on 11 May 1945 included Dr. Schneeberger, manager of the entire plant, Dr. Neumann, in charge of lubricating oil manufacture, and Dr. Billig, in charge of the chemical and engine-testing laboratory.

The plant is of conventional design and includes installations for atmospheric crude distillation, vacuum distillation of the atmospheric residue, propane deasphalting and deresining, phenol extraction and propane dewaxing, as well as conventional acid treating and clay contacting. The crude throughput was about 60,000 bbls/month.

A mixture of Hanover and Austrian crude was processed mostly, but more recently Austrian crude only was used. Typical data on two Austrian crudes are shown in Table I. The atmospheric distillation gave the following yields for overhead, three side-streams and bottoms, respectively:-

	Percent by volume of crude	Bbls/month
Gasoline....................................	10.6	6,350
Kerosine....................................	20.0	12,000
Spindle Oil.................................	16.7	10,000
Light Neutral dist. (about 130 SSU/100° F.)....	3.0	1,800
Residue.....................................	43.6	26,200
Loss..	6.1	3,650

Vacuum distillation of the atmospheric residue yielded the following distillates:-

Product	Percent by volume of crude	Bbls/month
Spindle Oil (60 SSU/100° F.)................	7.0	4,180
Heavy Neutral dist. (300 SSU/100° F. Min.)..	10.7	6,420
Residue..................................	26.0	15,600

The gasoline was not used for automotive purposes, but was utilized for special purposes (precipitation naphtha, petroleum ether, etc.). Kerosine was caustic washed and used for illumination; part of the kerosine was blended with spindle oil from the atmospheric distillation and the blend was used as diesel fuel. The remainder of the spindle oil was blended with the corresponding fraction from the vacuum distillation. Average inspection data on the distillation products are given in Table 2.

Finished neutral oils were made by dewaxing and acid and clay treatment of the 60 vis spindle oil and the light and heavy neutrals. Treating losses are given as follows:-

Charge stock	Percent by weight paraffin removed	Percent by weight acid sludge removed
Spindle oil 60 Vis.	20	4-5
Light neutral distillate..	20	4-5
Heavy neutral distillate..	20-25	4-5

The inspection data on the finished neutral oils are given in Table 3. The heavy neutral distillate was also used in the manufacture of automotive and aviation oils and for this purpose was subjected to phenol extraction instead of acid treatment. The yield of automotive oil was 70-75% by weight of the distillate, whereas the yield of aviation oil was 55-60% by weight. Inspection data of the finished oils are given in Table 4. The vacuum residue was propane de-asphalted and de-resined (yield of resin and asphalt free Oil: 50-60% by weight of vacuum residue charged). For the manufacture of automotive bright stock, the propane-treated vacuum residue was treated with 150 vols. of phenol yielding about 41% by weight of raffinate (calculated on vacuum residue charged). The raffinate was propane dewaxed and clay treated with an ultimate yield of automotive bright stock of about 28% by weight of the vacuum residue. In case that aviation bright stock was required, the raffinate from the phenol extraction was freed from phenol and again phenol-treated (total volume of phenol used in both stages: 500) yielding a raffinate which represented about 28% of the vacuum residue. The final yield of aviation bright stock after propane de-waxing and clay-treating was approximately 18.5% by weight of the vacuum residues. The inspection data for the two grades of bright stock are given in Table 5.

Propane deasphalting was carried out in a two-stage unit comprising 4 horizontal contactors and precipitated asphalts and resins were removed separately. Both of these products were destructively hydrogenated. The yield of deasphalted oil averaged 50% on charge.

Phenol extraction was carried out in a Kellogg unit built in 1938. It has a conventional tower type unit with perforated trays and water injection was employed to improve the selectivity of the solvent.

When automotive bright stocks were processed, the normal solvent ratio was 150%, the tower top temperature 90° C. and the temperature gradient 10° C. Aviation bright stocks, on the other hand, are solvent extracted twice, first under the above conditions, and then re-extracted with 500% of phenol at 110-120° C.

Dewaxing was carried out in a Dorr rotary pressure filter using discontinuous cooling. The charge stock was cooled from 86° F. to -40° F. in one hour. The design filtration rate was 7.5 gals. per cubic-foot per hour, but actually a rate of 19 gals. per cubic-foot per hour was obtained.

The working pressure was 150-200 mm. Hg. gauge. The double dilution technique was not employed and there was no re-pulping of the wax. The oil content of the finished wax averages 20%

LUBRICATING OIL SPECIFICATIONS;

Motor Oils

Three grades were produced having viscosities at 50° C. of 8, 10, and 12° E. and a maximum pole height of 2.24.

Aero Oils

The following specification was adhered to:-

S.G. @ 20° C.	<0.90
Conradson Carbon Residue	max. 0.3
Viscosity @ 50° C.	17° E.
" 100° C.	2.5° E.
Pole Height	max. 2.0
Pour Point	-17° F.

(No oxidation test)

It was stated that when production started at this refinery, a 100 ton batch of aero oil to this specification was prepared and, after engine builders had carried out acceptance tests with satisfactory results, regular production began and continued without interruption.

TABLE 1. - Austrian crudes (Average for October 1943)

Name of field	Hauskirchen	Neusiedel
Sp. gr. at 60°F.	0.870	.872
A.P.I. gravity	31.1	30.8
Engler Distillation		
ISP	248	248
10%	383	383
20%	482	480
30%	563	559
% at 572° F.	33	33
% water	1.8	1.8
% BS & W	2.2	3.6
% salt	0.046	0.019
Salt lbs/1,000 bbls	140	58
% ash	0.03	0.03
% paraffin	8.0	12.1
Solidification point of paraffin °F.	123	126
Neut. No.	0.12	0.18
% Sulfur	0.53	0.12

TABLE 2.

	Benzine	Kerosine	Atmos. Spindle Oil	Atmos. Neutral Oil	Atmos. Residue	Vacuum Heavy Spindle Oil	Vacuum Neutral Oil	Vacuum Long Residuum
Sp. Gravity at 60° F.	-	.824 - .844	.867	.886	.951	-	.903	.958
Octane No.	50	This material was soda washed and then sold to the railroads for use in signal lamps.	-	-	-	-	-	-
Viscosity °E.	Used for solvent manufacture but later sold as motor gasoline blended with hydro gasoline.							
20° C.			2.21	-	-	-	-	-
50° C.			-	2.85	26.7	-	4.82	-
100° C.			-	-	-	-	-	16.9
I.B.P. °F.			545	-	-	-	-	-
% at 572° F.			4	-	-	-	-	-
Flash (Open) °F.			280	383	356	-	437	563
Pout point °F.			39	66	84	-	102	95
Wax % weight			4.26	16.1	12.0	-	18.6	8.76
M.P. °F.			118	125	129	-	127	133
Asphalt				0.004	0.3	-	0.009	0.49
Viscosity, SSU								
100 °F.			about 60.0	about 130	about 2000		about 350	
210 °F.								about 600

TABLE 3. - Finished Neutral Oils

Distillate used in preparation of finished oil	Light neutral	Heavy neutral
Finished oil		
Sp. gravity at 60° F.	0.896-0.901	0.913-0.918
API gravity	25.5-26.5	22.6-23.5
Vis. at 50° C. °Engler	About 3.5-4.0	8.5-9.0
Vis. at 100° F. SSU	" 200	About 600
Pole height	" 2.15	2.4-2.5
Pour point °F.	Zero to 5° F.	-1 to -5° F.
Neut. No.	0.16	0.07
Sap. No.	0.22	0.17
Water %	0	0
Flash point, °F.	374	437

TABLE 4.

Distillate used in preparation of finished oil	Heavy neutral motor lube oil	Heavy neutral aviation lube oil
Finished oil		
Sp. gravity at 60° F.	0.891-0.901	0.881-0.886
API gravity	25.5-27.3	28.2-29.1
Vis. at 50° C. Engler.	6-7	5.5-6.0
Vis. at 100° F. SSU	About 440	About 350
Pole height	About 2.25	1.95-2.0
Pour point	Zero to -5° F.	Zero to -5° F.
Neut. No.	0.01-0.02	0.01-0.02
Sap No.	0.05	0.05
Water %	0	0
Flash point, °F.	428-437	446

TABLE 5. - Bright Stock from Vacuum Residue

	Automotive bright stock	Aviation bright stock
Sp. gravity at 60° F.	0.901-0.906	About 0.891-0.896
API gravity	24.7-25.5	26.4-27.3
Vis. at 50° C. °Engler	28-35	21-26
Vis. at 210° F. SSU	About 125	About 120
Pole height	2.25-2.3	1.95-1.99
Flash point, °F.	554-572	536-554
Pour point, °F.	Zero to -2.2	Zero
Conradson carbon residue.	--	0.3-0.4

OTHER PRODUCTS

Gasolines. Typical data - March 1942

	Hydro	Fischer-Tropsch
Sp. gr. @ 20° C.	0.749	0.700
I.B.P. °C.	41	40
5%	60	58
20%	79	74
50%	98	99
70%	113	127
90%	138	165
95%	150	183
F.B.P., °C.	154	192
Octane No. clear	73.5	56.5
V.P. atm.	0.66	0.32

Hydro gasoline was formerly of 180° C. end point, but this was reduced to 150-155° C. when the Hydro Gas Oil pour point specification was limited to -40° C. max. Fischer-Tropsch gasoline end point was later reduced for a similar reason, in this case the diesel oil cut being 160-320° C. of -12° C. pour point. Fischer-Tropsch Gasoline of 160° C. end point had a clear Octane Number of ca.60.

No aviation gasoline was produced at Lützkendorf.

DIESEL OILS

Fischer-Tropsch and hydro gas oils were despatched separately from Lützkendorf to various WIFO blending stations, while a blend of petroleum kerosine and atmospheric spindle oil was used internally in tractors and by local transport organizations.

	Hydro gas oil	Fischer-Tropsch gas oil
Sp. gr. @ 20° C.	0.865	0.743
I.B.P., °C.	179	173
20%	200	180
50%	224	195
70%	254	205
90%	328	254
Cloud point, °C.	-25	-38
Pour point, °C.	-41	-40
Filtration rate, secs.	38.4 @-28° C.	2.6 @-39° C.
Cetane number	35.6	68.2

Hydro diesel oil specification called for a maximum pour point of -40° C. and a Cetane Number of 40-45.

ENGINE TESTING

In the engine testing laboratory, the following engines were installed:-

I. G. Prüfmotor
C.F.R. Motor Method Engine
and Deutz Diesel Engine.

These were employed for routine Octane Number and Cetane Number determinations.

MERSOL MANUFACTURE

It was stated that a Fischer-Tropsch fraction b.p. 320 to 340° C. was despatched for Mersol manufacture, and that material boiling above 340° C. containing 10% wax, was sent to Witten for fatty acid manufacture.